Economical CO2, SOx and NOx Capture from Fossil Fuel Utilization with Combined Renewable Hydrogen Production and Large Scale Carbon Sequestration

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Abstract

The objective of this project was to investigate and demonstrate the methods of production at a continuous, bench-scale level and produce sufficient material for an initial evaluation of a potentially profitable method to produce bio-energy and sequester carbon. The novel process uses agricultural, forestry and waste biomass by producing hydrogen using pyrolysis and reforming technologies conducted in a 50 kg/hr pilot demonstration. The test runs produced a novel, nitrogen-enriched, slow-release, carbon-sequestering fertilizer. Seven kilograms of the material were produced for further plant growth response testing. A pyrolysis temperature profile was discovered that results in a carbon char with an affinity to capture CO2 through gas phase reaction with mixed nitrogen-carrying nutrient compounds within the pore structures of the carbon char. A bench scale project demonstrated a continuous process fluidized bed agglomerating process. The total amount of CO2 sequestration was managed by controlling particle discharge rates based on density. The patent pending process is particularly applicable to fossil fuel power plants as it also removes SOx and NOx, does not require energy intensive carbon dioxide separation and operates at ambient temperature and pressure. The method of sequestration uses existing farm fertilizer distribution infrastructure to deliver a carbon that is highly resistant to microbiological decomposition. The physical structure of carbon material provides framework for building a NPK fertilizer inside the pore structure and create a physical slow release mechanism of these nutrients. The complete process produces three times as much hydrogen as it consumes making it a net energy producer for the affiliated power plant.

http://www.eprida.com/hydro

Keywords: fertilizer, hydrogen production, direct carbon sequestration, profitable

Introduction

The increasing anthropogenic CO₂ emissions and possible global warming have challenged the United States and other countries to find new and better ways to meet the world's increasing needs for energy while, at the same time, reducing greenhouse gas emissions. The need for a renewable energy with little to zero emissions has lead to demonstration work in the production of hydrogen from biomass through steam reforming of pyrolysis gas and pyrolysis liquids. Our research to date has demonstrated the ability to produce hydrogen from biomass under stable conditions. A future of large-scale renewable hydrogen production using non-oxidative technologies will generate co-products in the form of a solid sequestered carbon. This char and carbon material represent a form of sequestered carbon that will not significantly decomposeⁱⁱ and return carbon dioxide into the atmosphere. A need was recognized that additional value could be added to this material that would justify large-scale handling and usage. Currently, carbon in the form of carbon dioxide is accumulating at the rate of 1.6 gigatons per year and increasing greenhouse gases by 1.5-3 ppm. The volume of waste and unused biomass economically available in the United States is over 314 gigatons per yearⁱⁱⁱ. Sequestering a small percentage could significantly reduce the atmospheric loading of carbon dioxide while producing a zero emissions fuel, hydrogen. In order to accomplish this economically, the sequestered carbon must have a very large and beneficial application such as use as a soil amendment and/or fertilizer.

Project Description

The approach^{iv} in our research applies a pyrolysis process that has been developed by Eprida and National Renewable Energy Laboratory (NREL) to produce char and synthetic gas (containing

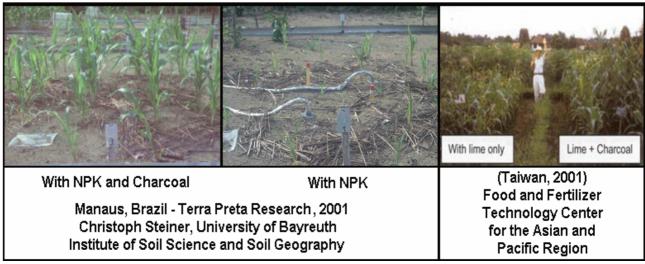
mainly H₂, and CO₂) from biomass, which could come from farm and forestry sources. In this novel system^v, the hydrogen is used to create ammonia and then combined back with the char and CO₂, at atmospheric pressure to form a nitrogen compound enriched char. The char materials produced in this process contains a significant amount of non-digestible carbons such as the elementary carbons that can be stored in soils also as sequestered carbon. Furthermore, the carbon in the char is in a partially activated state and is highly absorbent. Thus when used as a carrier for nitrogen compounds (such as NH₄⁺, urea or ammonium bicarbonate) and other plant nutrients it forms a slow-release fertilizer that is ideal for green plant growth. A combined NH₄HCO₃-char fertilizer is probably the best product that could maximally enhance sequestration of carbons into soils while providing slow-release nutrients for plant growth. Research work has shown^{vi} that char also provides the ability to capture farm chemical runoff. The verification of this product's capability as both a fertilizer and chemical sponge could lead to its use as an "Approved Management Practice" under the USDA Conservation Reserve Program; a pollution prevention program that provides farm payment for specific land management activities which reduce farm runoff pollution. The addition of a systematic technology concept viiviii developed recently at Oak Ridge National Labs could sequester industrial greenhouse gas emissions. This approach utilizes an innovative chemical process which can directly capture greenhouse gas emissions at the smokestacks by converting CO₂, NO_x, and SO_x emissions into valuable fertilizers (mainly NH₄HCO₃), which can potentially enhance sequestration of CO₂ into soil and subsoil earth layers, reduce NO₃⁻ contamination of groundwater, and stimulate photosynthetic fixation of CO₂ from the atmosphere. The inorganic carbon component (HCO₃) of the NH₄HCO₃ fertilizer is non-digestible to soil bacteria and thus can potentially be stored in certain soil and subsoil terrains as sequestered carbons. This technical approach integrates pollutant removal and fertilizer production reactions with coal fired

power plants and other energy producing operations, resulting in a clean energy system that is in harmony with the earth's ecosystem.^{ix} The key step in this technology is an NH₃-CO₂-H₂O reaction system to form solid NH₄HCO₃ process that can remove flue-gas CO₂ emissions through ammonia carbonation by formation of solid NH₄HCO₃ product. An important feature of this work to the power industry is that it does not require compressors or prior separation of the CO₂. Maximally, about 300 million tons of CO₂ per year (equivalent to about 5% of the CO₂ emissions from all coal-fired power plants in the world) from smokestacks can be solidified and placed into soil by the use of this technology. The combination of these two novel approaches offer an opportunity for fossil energy systems, farmers, and the fertilizer industry infrastructure to become the large contributors to meeting Kyoto greenhouse gas reduction targets.

The goal of our research was the laboratory and pilot scale demonstration of a sequestering fertilizer, with properties which could increase crop yields, soil carbon content, water holding capacity, nutrient retention, cation exchange capacity and microbial activity while decreasing farm chemical runoff, nutrient leaching, and greenhouse gas emissions. The advantages of an adsorbent charcoal provided many of the characteristics we sought and creating a material that farmers could rely on to slowly release imbedded nutrients continuously to the crops or forest during the growing season was one of our first development goals.

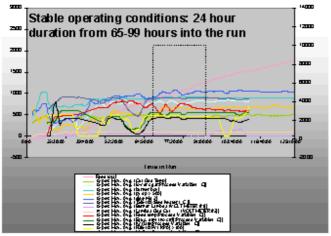
Adding nutrients to soils does not mean that they become available for plant growth^x. Nutrients can be leached from the soil, they can bind with clay materials reducing availability, or escape through atmospheric interactions. The first goal was to identify process parameters that would produce a carbon material that could act as a nutrient carrier and would resist leaching. It appeared that charcoal addition from even 2000 years ago was providing significant soil fertility

benefits xi in the research conducted on terra preta soils by Glaser, Lehman, Steiner and the addition of charcoal to the soil xii .



xiii

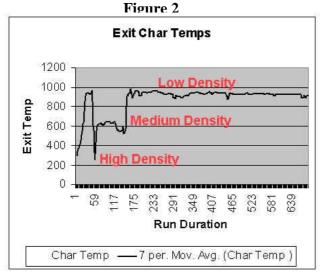
Figure 1



We first began our investigation by looking at charcoals made under different conditions. We had made a number of types of char during a 100-hour hydrogen demonstration experiment conducted in the summer of 2002^{xiv}. The goal during the run was to produce hydrogen with a co-

product.

The co-product char was highly dependant on processing conditions. As can be seen from the chart, our start up phase had significant variations in operating conditions. The changes in gas



flows, feed rates and heat rates eventually smoothed out to stable run conditions as we tweaked process parameters. However, these changes in process gave us an opportunity to examine the materials that were being made.

After the run, we measured the density

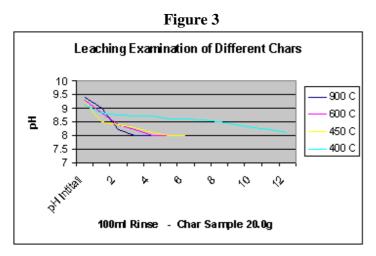
of each material stored in the sealed 55-gallon drums. Each barrel had been labeled with a date and time so that we could match it up with the corresponding production data. The first physical measuring of density in each barrels char gave us 3 distinct materials. Most of the char was a low density, material produced during the long stabilized run conditions for the hydrogen experiment. The high-density material represented only a small portion of the total and due to multiple variations in process conditions during that time pinpointing any specific set of parameters proved difficult.

At this point we decided to see if there were any attributes other than density that made these three materials different. We ground 40 grams of each material to 30 mesh, making a small grainy powder and then added two grams to 50ml water. In both the high and medium density chars, the powders immediately sank to the bottom of the flask. The low density floated and had to be stirred vigorously before it sank. It appeared that the open structures of the higher temperature char had no resistance to water at all.

We conducted bench scale experiments to reproduce these materials under precise controls so we could accurately determine temperature and conditions which created the materials and the effects on the performance of the material as a nutrient carrier. We produced 5 different chars, at different temperatures (900C, 600C, 500C, 450C and 400C). A metal 560ml stainless steel can with a press-in top had a 6mm hole drilled in the lid. A 6.4mm stainless tube 10cm long was tapped in to fit firmly in the hole as an exhaust port. The biomass samples (peanut hull pellets) were weighed and placed in the can and the top sealed. A Thermolyne model 1400 box furnace was preheated to each temperature for 10 minutes before the stainless steel container was inserted. The exhaust tube was fed out through a 75mm port in the back of the furnace. An external thermocouple inserted into the free space between the can and the wall of the furnace interior operated a separate controller to give precise control of the temperature experienced by each sample. Within a few minutes after placing the container in the furnace, the pyrolysis vapors began to escape. At 10 min intervals, a small 1.5mm thermocouple was inserted through the exhaust tube and a temperature of the material taken directly. After several experiments, we were able to gauge that until the high volatile gas evolution slowed, the readings would not exceed 350 degrees in the material. So we changed our method and began taking internal sample temperatures after the gas flows had slowed to minimal amounts, generally around 370-380 degrees. Once the temperature was within 50 degrees, the thermocouple would be left in the sample. In each case the samples were brought to the target temperature for 1 minute.

After reaching the target temperature, we removed the container from the furnace and turned it upside down on a smooth surface metal table to cool. We found that the material still evolves some CO2 and with the small hole, no oxygen can get to it until it has cooled to a point where it will no longer oxidize. All our samples were produced with this technique. Next the materials

were ground by hand and sieved to a particle size less than 30 US mesh and greater than 45 US mesh and 20.0-gram samples were prepared. We mixed an aqueous solution of 48% NH₄NO₃ (ammonium nitrate). Each sample was soaked for 5 minutes and then poured through cone filter paper and allowed to air dry for 24 hours. We then poured rinses of 100 ml of water (pH8) through the cone filter and measured the pH of each resulting rinse and measured a decreasing pH commensurate with the leaching rates of each material.



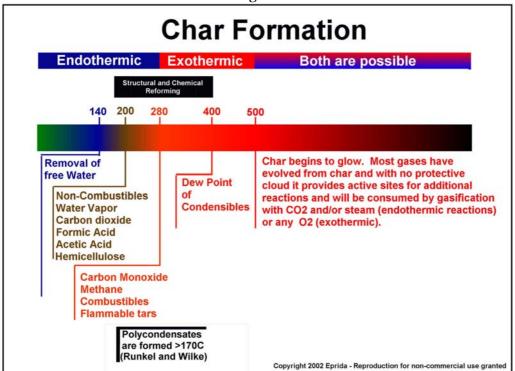
In these experiments there was very little difference until the last one. After three or four rinses the materials would stabilize at the pH 8 of the rinse material. The 400C char showed very little change and it was only after the 9th rinse that it began to drop a bit

faster but even after 12 rinses it still had not stabilized. It looked like a good candidate for further testing.

The material could be considered comparable to those that have been made in a smoldering forest fire. Chars have been found to support microbial communities. **V* The breakdown of plant matter, the adsorption of these nutrients by a layer of char below and a niche for microbes to grow, Pietikainen suggests are the reason for the success of microbial communities in char in her study. However, char exposed to high intensity fire and temperature, as we have seen above, may adsorb but may not provide the same levels of retention that could offer a superior material for long term slow release of nutrients.

If the hypothesis is that we want to adsorb, store (reduce leaching effects) and yet provide a safe haven and an environment for microbial communities to flourish, then investigating the science of char production may help.

Figure 4



An illustrative chart shows properties of char formation, which can vary according to the composition of the originating biomass. In this chart, the material is shown entering a phase from 280 to 500 °C that is exothermic. Once started, it continues without additional heat. If oxygen is present or if the material is left in its exothermic environment it will continue past the structural and chemical reforming zone and become normal char. In certain temperature ranges of pyrolysis, reactive low molecular weight products will further react to form polycondensates^{xvi}, which will eventually volatilize and leave the char as the temperature increases. The deposition of condensables in a char bed is well known and generally the issue

has been how to keep these materials from building up on downstream process. The design of our reactor was developed specifically for this reason. However, intra-particle condensation leads to increased char mass and a modification of the surface structures. The deposition of these materials may increase microbial activity. xvii

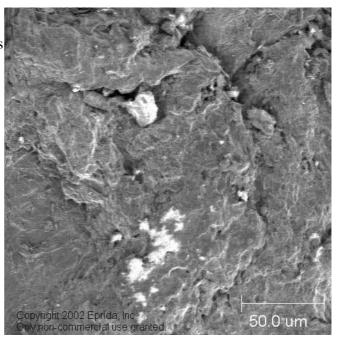
SEM Investigation of Char and Enriched Carbon, Organic Slow release Sequestering (ECOSS) Fertilizer Figure 5

Figure 5 is a scanning electron microscope image (SEM) of the 400C char. This picture is taken at 70x. As you can see in the bottom right hand corner and the image below, the physical structure of the cellulose material is apparent.

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The char particles are a hard carbon with more strength the higher temperature chars, but grinds easily. These particles are hydrophobic in nature and differ from char made at high temperatures.

Figure 6



The next image is taken at 350x and provides more detail of the char's surface. The image reflects some of the biomass origin as the physical structure had been broken up by the mechanical actions of pelletizing. Its surface shows evidence of the cellulose layers. The internal gases that escape from the material during the charring help develop charcoals natural porosity. But at this level of magnification, the pore structure is not visible.

Figure 7

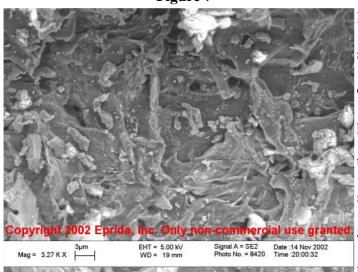
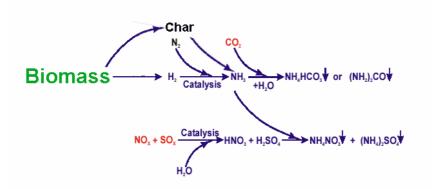


Figure 7 is taken at 3270x clearly showing pore structures. The evolution of this adsorbent material provides a porous internal structure as well. Using the parameters established in the bench scale char production, we selected 400 C as the target temperature for the char to reach before being discharged from our

pyrolysis reactor. The material was fed into the pre-heated reactor and the gases that evolved were flared. No external heat source was needed and the materials rapidly moved into an exothermic reaction. The continuous process system was automatically fed material by a level indicator and discharged into a nitrogen purged 55-gallon drum upon reaching 400°C. The resulting char was cooled for 24 hours then fed through a two-roll crusher and then sieved with a mechanized screen through 30 mesh and 45 mesh screens. The resulting fraction remaining above the smaller screen was chosen as our starting material.

The technology developed at the Oak Ridge National Laboratory by James Lee and his team has recently demonstrated that removal of flue-gas CO₂ can be achieved via formation of solid

Figure 8

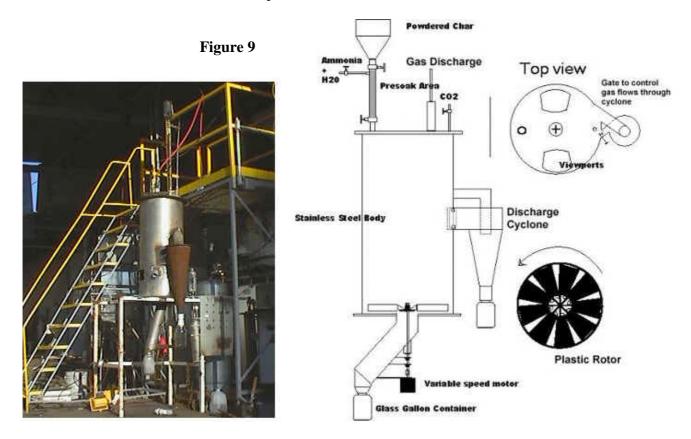


NH₄HCO₃ through ammonia carbonation in the gas phase. This result indicated that it is possible to use NH₃-CO₂-solidifying technology to remove

greenhouse-gas emissions from industrial facilities such as a coal-fired power plant. A joint ORNL and Eprida, Inc. study of a specifically designed char materials produced by Eprida, Inc. from a biomass pyrolysis process, indicated that it might also be possible to use those char particleshad demonstrated the physical solidification of CO2 for sequestration of power plant greenhouse gas exhaust. It was assumed that the char could potentially be used as a catalyst (providing more effective nucleation sites) to speed up the formation of solid NH₄HCO₃ particles in the NH₃-CO₂-solidifying NH₄HCO₃ production process, thus potentially enhancing the efficiency of the NH₃-CO₂-solidifying technology. Furthermore, the integrated process technology could produce a valuable NH₄HCO₃-char product that could maximally enhance sequestration of carbons into soils and remove SOx and NOx while providing an ideal "enriched carbon, organic slow release sequestering" ("ECOSS") fertilizer nutrients for plant growth if the production of NH₄HCO₃ could be developed inside the porous carbon media.

The question of whether the NH₄HCO₃ fertilizer could be created in the internal pore structures in an environment similar to what might be produced in an industrial environment required an experimental setup. A preliminary design was built. While the process can apply to many configurations, the development of a simple production technique was important at this stage in our research efforts and commercial implementation.

In this case we used a mechanical fluidized bed easily adaptable to any gas stream and injected CO2, ammonia saturated with water vapor.



A 250-gram charge of 30-40 mesh 400C char was fed in at regular intervals varying from 15-30 minutes. A higher rotor speed increased the fluidization and suspended the particles until they became too heavy from the deposition of ammonium bicarbonate to be supported by fluidized gas flows. The longer durations produced significantly larger particles.

Figure 10

The image on the left

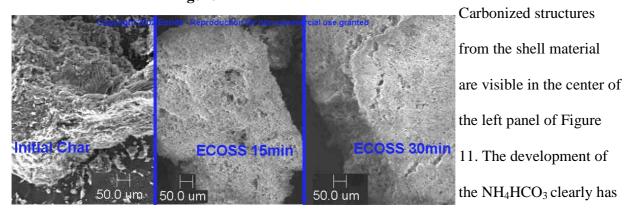
Char after H2 extraction

Char ECOSS - Sand like

Large Granule ECOSS

in Figure 10 is the initial char. The image in the center is a material produced after 15 minutes and the one on the right after 30 minutes.

Figure 11



coated the char, but the next analysis was to determine reactions inside the carbon structures. We crushed the ECOSS-15 minute material to see inside.

A SEM Investigation of the Interior of an ECOSS-15 Char Particle

Figure 12 is a char particle at 422x. It has been crushed and a chunk was broken off in the process. It appears that the Ammonium Bicarbonate formed in the fractured areas between the main body of the particle and the broken chunk.

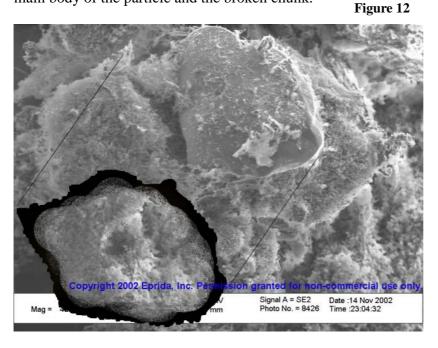


Figure 12 shows a simulated image of the part of the char that was broken off.

Figure 13

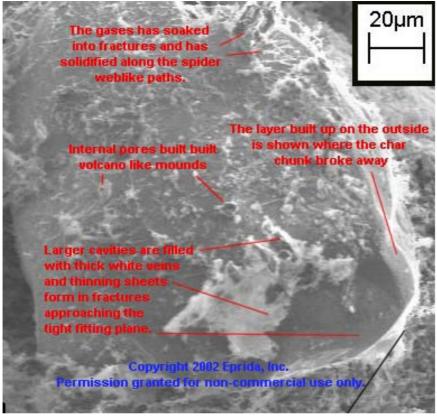


Figure 13 shows where the verv small molecule ammonia was adsorbed into the char fractures and internal cavities. As carbon dioxide enters, it converts the ammonia into the solid, trapping it inside the micro-porous material. The internal flat-top volcano like structures demonstrate the impact of

ammonia and carbon dioxide meeting in a pore where the ammonia bicarbonate builds up on the inside, closing the larger pore from the inside.

Figure 14

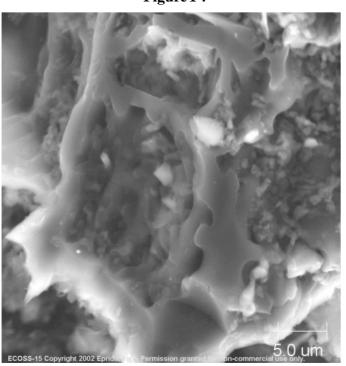


Figure 14 is a closer look at the crushed material (2000x); we can see small deposits beginning to form inside the carbon framework. There appear to be plenty of open spaces for microbial interaction. Additionally, reformation of polycondensates on the carbon structures may contain nutrient sources for enhancing microbial activity.

Figure 15

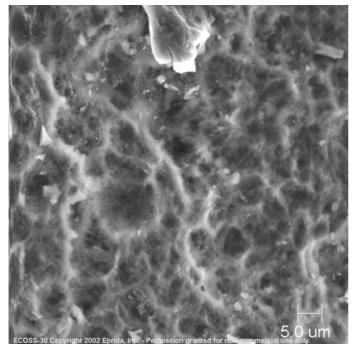


Figure 15 is a 1000x image of the interior of the ECOSS produced after 30 minutes. The SEM shows that the interior of the char is filled with the fibrous NH₄HCO₃ deposits. A column of carbon is shown broken off at the top of the picture.

The exact duration for ultimate delivery

and release of nutrients solidified deep in these internal pore structures need further testing both in bench scale and soil plot testing. Yet, the simple physical solidification of nutrient material and sequestered carbon dioxide inside the char structure can be seen. This demonstration of the process shows that we can deposit nutrients inside the porous media.

Production Chemistry Calculations and Carbon Utilization

Calculating the amounts of ECOSS and hydrogen that can be made from a standalone system would begin with a typical initial biomass composition of: xx

C 46.0%

H 6.3%

O 42.5%

N 2.2%

Ash 3.0%.

Yield of charcoal is 32% on dry biomass basis (ash and nitrogen are totally sequestered in charcoal). Typical composition on ash and nitrogen free basis is

C 82%

H 3.4%

O 14.6%.

While charcoal production may go up or down, the above number reflects a reasonable dry weight estimate.

ECOSS (at 10% Nitrogen Rates) is a mixture of 56.4% NH4HCO3 (at 17.7%N so therefore the end ratio nets 0.1/0.177) and 43.6% charcoal. This actually produces a 12%+ N fertilizer as char has approximately 2 - 4 % N trapped in the char. However for the purposes of this analysis it is not utilized as availability and rate of release of this N have not been verified.

Based on the above, from 100 kg of biomass you can produce (32/43.6)*100 = 73.4 kg ECOSS for which you need 41.4 kg NH4HCO3. For 1 mole (79g) NH4HCO3 you need 5g of hydrogen, and for 41.4 kg you will need (5/79)*41.4 = 2.64kg hydrogen

By pyrolysis of 100 kg biomass you will produce 32 kg charcoal and 68 kg gas and vapor.

Considering the composition of charcoal the elemental balance will be as follows: xxi

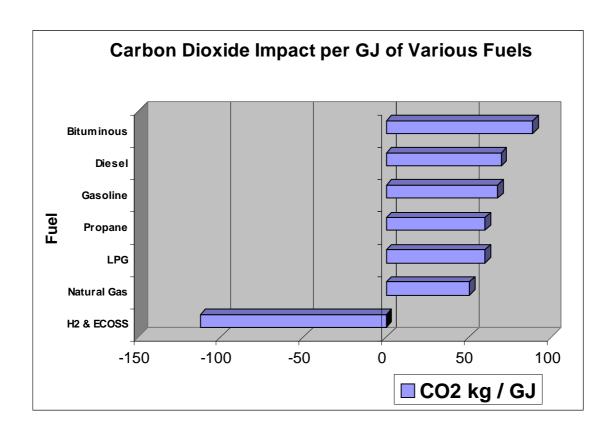
	In		Out
100 kg of biomass		32 kg of charcoal	68 kg of gas + vapor
C	46	22	24
Н	6.3	1.1	5.2
O	42.5	3.7	38.8
N	2.2	2.2	
ash	3.0	3.0	

The 22kg of C represents a the stable form or sequestered carbon we use as the framework for building a new type of fertilizer.

Reforming (assuming total conversion of carbon to CO2) will require (stoichiometrically) 28.35 kg steam and will produce 88 kg CO2 and 8.35 kg hydrogen. Because only 2.64 kg H2 is necessary for ECOSS (using all charcoal available from the process) 6.78 kg (68.4% total production) of hydrogen will remain per every 100 kg of biomass.

The amount of C, converted from atmospheric CO2 is equal to 12kg for each 79kg of ABC or 15.2% sequestered carbon as a ammonium bicarbonate. Therefore for each 100kg of biomass, we will produce (12/79)*41.4 kg or an additional 6.29 kg of C or a total of 28.3kg of utilized carbon. In acid soils this part of the carbon will convert to CO2 but in alkaline soils it will mineralize and remain stable. According to USDA reports, 60-70% of worldwide farmland is alkaline, so conservatively allowing for 50% of the bicarbonate to convert to CO2, this will leave us with approximately 25kg of stable carbon in our soils for each 100kg of biomass processed. This carbon represents equals 91.5kg of CO2

A different way to look at this is to compare the amount of energy produced and the resulting CO2 impact. With 6.78kg hydrogen extra produced per 100kg of biomass, then 25/6.78 = 3.69kgC/ kg H or 3.66x3.69=13.5kg CO2 / kg of hydrogen produced and used for energy. From a power perspective, that is 13.5kg CO2 / 120,000 KJ of hydrogen consumed as a renewable energy or 112kg/GJ of utilized and stored CO2.



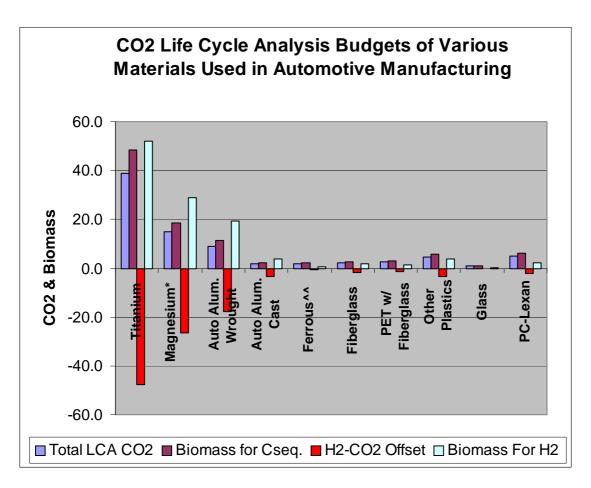
Global Potential

The large majority of increases in CO2 will come from developing countries as their burgeoning entrepreneurial populations industrialize. A sustainable technology needs to be able to scale to meet the growing needs of this large segment of the population. Developing an economical size that offers a profitable platform may require certain minimums and it may be that the lower limit of economical production are larger than typical biomass conversion systems. A 1-2MW facility could be the lower limit yet there are two factors that are important to note. The first is that the low relative efficiencies required by both the hydrogen separation and the ammonia production may allow a smaller foot print system to be developed using new technologies. Future research

efforts in separations technologies and ammonia catalyst could offer developments that lead to systems for even very small farming communities.

The second point is that the total hydrogen is approximately three times the maximum that can be utilized in one facility, so every third facility could be designed to accept the charcoal that is produced by two standalone energy systems. This special facility could process all of its hydrogen and the carbon from two other locations and use existing industrial ammonia manufacturing techniques to create the carbon-fertilizer. If all hydrogen is converted to fertilizer then there is an opportunity to acquire outside CO2 (34kg required for each 100kg biomass processed) and the opportunity to earn revenue from SOX, NOX removal could provide it with another income stream and help its economics. It would also fit closely into strategies of developing areas that wish to attract and support GHG emitting manufacturing.

The energy from a total systems point of view could create a carbon negative energy as detailed in the IIASA focus on Bioenergy Utilisation with CO2 Capture and Sequestration (BECS). The effects shown in the prior graph (ie. providing 112kg of CO2 removal for each GJ of energy used) could allow major manufacturers to offset their carbon costs. The graph below shows various materials used in automobile manufacturing and the life cycle analysis on carbon emissions per kilogram. The second bar in purple represents the amount of biomass, using this process, that would be required to offset that carbon cost. The third bar in red shows the amount of sequestered carbon that would be created if the process were used to produce all the energy required for production and the last bar represents the amount of biomass required to meet the energy needs of producing that amount of material. In some examples the amounts needed for energy production are less than the amounts needed for carbon offset.



(ORNL-2002)

The opportunity for economically developing areas with biomass is to utilize their resources to help manufacturers reach carbon-negative status. If the material leaves a factory with a net carbon negative budget, then the behavior of consumerism becomes an agent of climate mitigation and supports economies in side stepping fossil fuel pathways.

How large could this method be applied and to what areas of the earth could utilize a concerted effort to reclaim eroded land? The positive impact of an increased soil carbon content ultimately

leads to increased food and plant yields, further helping to reduce CO2 buildup. There is very little information on the maximum rates of utilization, though 10,000kg/ha of char have been used with very positive results. Estimating the impacts on existing energy infrastructure shows that shift what way are doing could make a huge difference.

From above we saw that 1Gj of hydrogen produced and used will represent 112kg of utilized and stored carbon dioxide. Therefore, taking the atmospheric rise of 6.1GT and dividing by 112kg/Gj = 54.5EJ. This number falls amazingly along the 55EJ estimate of the current amount of biomass that is used for energy in the world today. While the potential reaches many times this for the future utilization of biomass, this shows that there is a chance that we can be proactive in our approach.

This concepts of biomass energy production with carbon utilization opens the door to millions of tons of CO2 being removed from industrial emissions while being used as an agricultural commodity, soil amendment and remediation product simultaneously producing a zero emissions fuel that can be used to operate farm machinery and provide electricity for rural users, agricultural irrigation pumps, and rural industrial parks. Future developments from the global research community will produce a range of value added carbon containing co-products from biomass. With the development and future use of this invention, both the producer of carbon dioxide and agricultural community have the capability to become a significant solution to the global rise in greenhouse gas emissions while building a sustainable economic development program for rural and agricultural areas.

About Eprida

Eprida, Inc. is a for-benefit company. For-benefits are a new class of organization. They are driven by a social purpose, they are economically self-sustaining, and they seek to internalize their social costs by being socially, ethically, and environmentally responsible. Closely associated is the Eprida Foundation, which has been established to take novel ideas and technologies on royalty free basis to economically developing areas and to assist in providing business model development and support for entrepreneurial efforts, with a mission to "Think like the planet".

Acknowlegements: Tyler Day, who has foregone many play days with Dad to allow time for writing. Stefan Czernik, who patiently helped me organize the chemical mass balances.

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